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Acquisition of Hyperthermal Atomic Oxygen Source

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6. AUTHOR(S)

Judith C. Yang

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

University of Pittsburgh
3700 O'Hara St.
Materials Science and Engineering
848 Benedum Hall
Pittsburgh, PA 15261

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)

Maj. Jennifer Gresham, AFOSR
4015 Wilson Blvd, Room 713
Arlington, VA 22203

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13. ABSTRACT ***(Maximum 200 Words)***

The objective of this grant was the acquisition of a hyperthermal atomic oxygen (AO) source that is of critical importance to a Multi-University Research Initiative (MURI) on the fundamental mechanisms of materials degradation in the low earth orbit (LEO). ("Simulations of Fundamental Degradation Process in Space Environments", PI: J. C. Yang, MURI Contract #: F49620-01-1-0336). A Physical Sciences, Inc. (PSI) FAST™ (Fast Atom Sample Tester) source was specially designed for portability with an ultra-high vacuum exposure chamber and delivered to University of Pittsburgh in October 2003. The portability of this FAST™ AO source allows the utilization of unique experimental tools located at different sites. This system has now been improved so that the best base vacuum is 4×10^{-9} torr. A room temperature sample stage has been built, a dual head quartz crystal monitor (QCM) system has been installed for *in situ* weight loss. A sensitive Mettler balance was purchased in order to measure total weight changes. All of these items have been installed and working since May 2004. Present experiments are being performed on Si, Al, Ag and Au single crystals and thin films.

14. SUBJECT TERMS	15. NUMBER OF PAGES
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17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT

Defense University Research Instrumentation Program
DURIP

ACQUISITION OF HYPERTHERMAL ATOMIC OXYGEN SOURCE

FINAL REPORT
AUGUST 11, 2004

Principal Investigator: Judith C. Yang, University of Pittsburgh

Address: Materials Science and Engineering Department
848 Benedum Hall
3700 O'Hara St.
University of Pittsburgh
Pittsburgh, PA 15213
(412) 624-8613
FAX: (412) 624-8069
Jyang@engr.pitt.edu

DURIP Grant Number: F49620-02-1- 0198

Program Manger:
Lt. Col. Paul Trulove (past), AFOSR
Maj. Jennifer Gresham, PhD (present, since 8/2004), AFOSR

Start Date: May 1, 2002

A. OBJECTIVES

The objective of this grant was the acquisition of a portable hyperthermal atomic oxygen (AO) source that is of critical importance to a Multi-University Research Initiative (MURI) on the fundamental mechanisms of materials degradation in the low earth orbit (LEO). ("Simulations of Fundamental Degradation Process in Space Environments", PI: J. C. Yang, MURI Contract #: F49620-01-1-0336, Program Manager: Lt. Col. Paul Trulove, AFOSR, present program manger: Maj. J. Gresham, AFOSR). Hyperthermal atomic oxygen is the primary species in the low earth orbit that rapidly degrades materials used in space vehicles. The Physical Sciences, Inc. (PSI) FAST^{TM} (Fast Atom Sample Tester) sources are the only hyperthermal atomic oxygen sources capable of continuous operation at both the appropriate orbital velocity and high flux. This source employs a laser-supported detonation wave heating of pure molecular oxygen to generate atomic oxygen continuously with kinetic energy of 5 eV with high flux. Physical Sciences, Inc. (PSI) (contact: Bob Krech) specially designed and manufactured a FAST^{TM} AO source, that was portable (on wheels), contained an ultra-high vacuum (UHV) chamber, and capable of very low dose for monolayer oxidation studies. This FAST^{TM} AO source was delivered to University of Pittsburgh in October 2003.

The portability of this FAST^{TM} AO source allows the utilization of unique experimental tools located at different sites. Specifically, ultra-high vacuum scanning transmission electron microscopy (UHV-STEM) and Surface Science Center, located at University of Pittsburgh, and synchrotron X-ray Diffraction (XRD), located at Brookhaven National Laboratory and Argonne National Laboratory.

A. STATUS OF EFFORT

The negotiation between PSI and the University of Pittsburgh took several months to clarify issues such as warranty (PSI finally agreed to a 1 year warranty), training, final demonstrated performance, and delivery costs. The Pittsburgh FAST^{TM} AO source was designed, built, and delivered in October 2003 by Bob Krech, where installation and training was conducted at Pittsburgh. The Pittsburgh FAST^{TM} 5 eV atomic oxygen system includes a cryopumped stainless steel vacuum test chamber with a pulsed nozzle assembly, a radiometric photomultiplier based beam velocity measurement system, a laser beam focusing system, control electronics, and a CO_2 laser. The primary FAST^{TM} AO Source specifications are listed in Table 1.

Fast O-Beam Properties	
Velocity	8 km/s ± 15% (5 to 12 km/s range)
Fluence	~10^{18} O-atoms/pulse, 3 Hz
Composition	>80% oxygen atoms
Size	Expandable to >1,000 cm^2 area
Charge content	<1% ions (controllable by pseudo-Helmholtz coils
Metastable content	$O(^1D)$ concentration <0.4%
Temperature	T = 300 K
VUV/UV content	One photon per 10^4 O atoms (similar to LEO)

Table 1. Summary highlights the properties of the oxygen beam.

The base vacuum of the sample chamber of the AO source after delivery was about 10^{-5} torr, in part due to a complication with the turbo pump control system. The Pittsburgh FAST^{TM} AO source was also missing some items that were necessary to use it, such as a sample stage, bake-out system (since it was designed to be UHV), and a system to measure the fluence and species within the beam. The vacuum system and monitoring system has now been improved so that the best base vacuum is now $4x10^{-9}$ torr. Furthermore, a room temperature sample stage has been built, a dual head quartz crystal

monitor (QCM) system has been designed and purchased and a very sensitive Mettler balance has been purchased to measure weight changes. The QCM system was designed to measure weight loss of thin films, such as Ag or C, that are standard materials to calibrate the AO fluence for very small AO doses. The Mettler balance was purchased in order to measure weight changes of Kapton, which is a standard witness material to determine total AO fluence. All of these items have been installed and working since May 2004. Table 2 is the list of items that were purchased, including the original cost of the FAST™ AO source, that was covered by the DURIP award:

<table>
<tr><th>No.</th><th>P.O.#
Date</th><th>Category</th><th colspan="2">Description</th><th>Vendor</th><th>Prices ($)</th></tr>
<tr><td>1</td><td>94863
10/28/2003</td><td>FAST AO</td><td colspan="2">Compact style.</td><td>PSI</td><td>350,000</td></tr>
<tr><td>2</td><td></td><td>Power Supply Cable</td><td colspan="2">For AO source power supply, NEMA type</td><td>Made at Pitt</td><td>200</td></tr>
<tr><td rowspan="4">3</td><td rowspan="4">456202</td><td rowspan="4">4 Regulators and gases</td><td>O_2,</td><td rowspan="4">With regulators $275each</td><td rowspan="4">Penn Oxygen</td><td>446</td></tr>
<tr><td>N_2</td><td>311</td></tr>
<tr><td>Laser mix75</td><td>475</td></tr>
<tr><td>He</td><td>402</td></tr>
<tr><td>4</td><td>467861</td><td>Hazard gas detector</td><td colspan="2">Model HIC, for CO, etc.</td><td>M[c]. Master-Carr</td><td>177</td></tr>
<tr><td>5</td><td></td><td>Sample stage</td><td colspan="2">Room temperature</td><td>Made in Pitt machine shop</td><td>200</td></tr>
<tr><td>6</td><td>473267</td><td>High vacuum gauge with controller</td><td colspan="2">MKS 943, 10^{-10} to 10^{-2} Torr range</td><td>Kurt J. Lesker Company</td><td>1,095</td></tr>
<tr><td>7</td><td>490371
05/06/2004</td><td>QCM(quartz crystal microbalance) sensor with RQCM controller</td><td colspan="2">Dual bake-able sensor heads, RQCM controller with sensor crystals</td><td>Maxtek, Inc.</td><td>7,484</td></tr>
<tr><td>8</td><td>494735</td><td>PC computer</td><td colspan="2">For RQCM</td><td>Dell</td><td>1,336</td></tr>
<tr><td>9</td><td>509179</td><td>Internal bake-out heater (IR halogen quartz lamp) with controller</td><td colspan="2">IR-123-120</td><td>Huntington Mechanical Labs. Inc.</td><td>1,686</td></tr>
<tr><td>10</td><td>105073
02/16/2004</td><td>Microbalance</td><td colspan="2">Mettler Toledo MX5, Readability 0.01 mg</td><td>Fisher Sci.</td><td>11,185</td></tr>
</table>

TOTAL:	**$374,997**
DURIP award:	$350,000
Pitt contribution through MURI-AFOSR award:	$24,997

Table 2: Summary of items and costs for the Pittsburgh FAST AO source.

The fluence as determined by both the weight loss of the Kapton witness sample and Ag weight gain as monitored by the QCM system was approximately 4×10^{19} AO/cm^2 and 2×10^{19} AO/cm^2, respectively for 50,000 shots or approximate 6×10^{14} AO/cm^2/shot where the distance between the nozzle and sample stage was 30 cm. This dose can be altered by varying the distance between the sample and the nozzle. Figure 1 is an image of the Pittsburgh FAST AO source, and Figure 2 is a close-up image of the ultra-high vacuum chamber for this source.

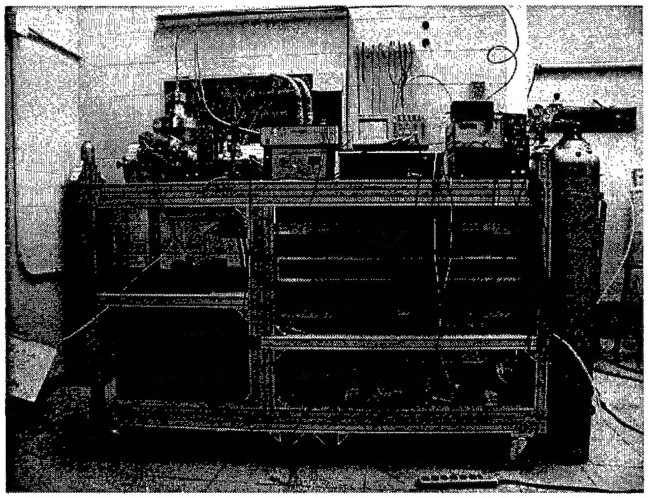

Figure 1: Pittsburgh FAST AO source.

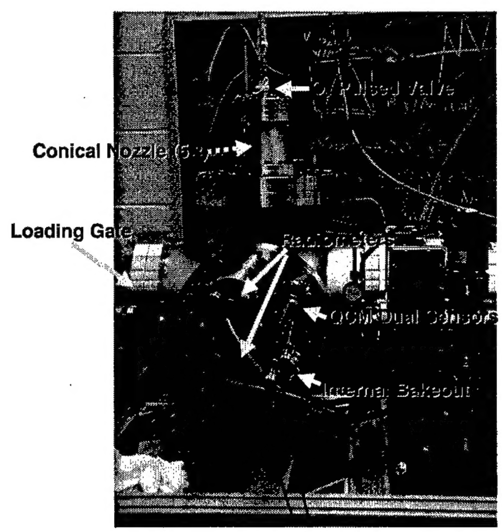

Figure 2: Image of the UHV chamber of the Pittsburgh FAST AO source.

C. ACCOMPLISHMENTS

Thin films of Ag, Al and Au were coated onto the quartz crystal and the weight change was monitored by the QCM system. Au revealed no weight change, whereas Al showed some weight increase and Ag showed a significant weight increase (Figure 3). From previous electron microscopy investigations, it is known that the "oxide scale" on Ag due to AO exposure is mostly polycrystalline, thereby demonstrating that Ag does not oxidize but continuously absorbs oxygen, which explains the initial linear increase of weight of the Ag when exposed to atomic oxygen.

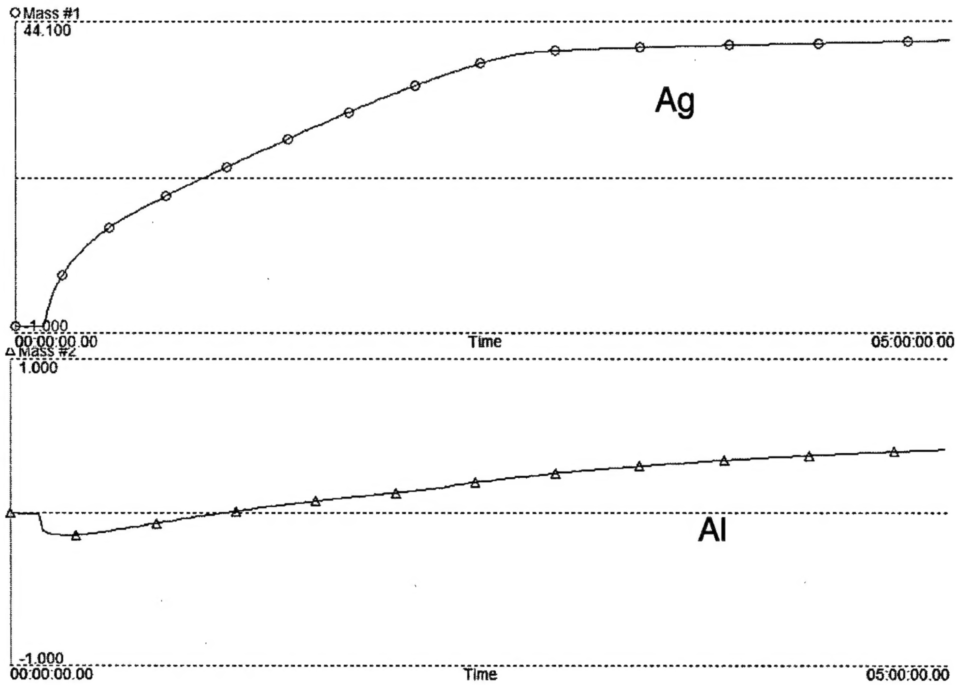

Figure 3: Weight gain of Ag and Al thin films when exposed to hyperthermal atomic oxygen within the Pittsburgh FAST AO source, as monitored by a dual-head quartz crystal monitor system. The total AO fluence is calibrated, using both a Kapton witness sample and Ag film, to be $3x10^{19}$ AO/cm^2 for 50,000 shots of the CO_2 laser. Silver shows a rapid increase in weight, whereas aluminum shows only a little increase in weight.

D. PERSONNEL

Judith Yang, Associate Professor, Materials Science and Engineering, University of Pittsburgh

Ian Robinson, Professor, Physics, University of Illinois at Urbana-Champaign

John T. Yates, Jr., Professor, Chemistry, University of Pittsburgh

Long Li, Research Associate, Materials Science and Engineering, University of Pittsburgh

Ross Harder, Post-doc, Physics, University of Illinois at Urbana-Champaign

Maja Kisa, Graduate Student, Materials Science and Engineering, University of Pittsburgh

E. PUBLICATIONS

None to report at this time.

F. INTERACTIONS

F. 1. Conference Presentations

"Different Amorphous Oxide Structures Formed by Oxidation of Si and Al in Reactive Oxygen Species", Invited Talk, Speaker: Judith Yang, Microscopy and Microanalysis, 4 August 2004, Savannah, GA.

F. 2. Consultative Function

1. Comparison between atomic oxygen sources at Montana State University and at University of Pittsburgh.

2. Development of Materials International Space Station Experiment (MISSE) 6 mission (includes AFOSR and Boeing funding) where identical samples will be exposed to the atomic oxygen in the low earth orbit and compared to the FAST AO source to determine how well the ground-based AO source compares to the space environment.

G. PATENTS

None to report at this time.

H. HONORS & AWARDS

Ian Robinson (Physics, University of Illinois at Urbana-Champaign) is a Fellow of the American Physical Society and the 2000 winner of the B. E. Warren Diffraction Physics Award of the American Crystallographic Association.

John T. Yates, Jr. is a R. K. Mellon Professor of Chemistry, University of Pittsburgh since 1981. He is a Member of the National Academy of Sciences (1996), the recipient of the von Humboldt Senior Scholar (1995-1997) and the J. Linnett Professorship from Cambridge University (2000).

Judith C. Yang (Materials Science and Engineering, University of Pittsburgh) is the 2002 recipient of a NSF Career Award on fundamental oxidation investigations.